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Wavelets image analysis for Friction Stir Processed TiNi functional behavior characterization

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Abstract

A key topic regarding Ti Ni Shape Memory materials concerns the possibility to attain welded junctions that preserves the shape memory properties of material. Other research topic for SMAs regards the retention of the shape memory effect cyclic stability; in fact, good shape memory properties frequently decrease during SME cycling of material. A method able to improve the cyclic stability of TiNi shape memory effect is the grain refinement. Considering these above mentioned research topics, a solid state welding process, as the Friction Stir Welding, is thus attractive for SMA joining and it exhibits potentials for achieving welded joints affected by microstructural changes that preserve the shape memory properties and retain, furthermore, the cyclic stability of SME. The basic objective of this study was to investigate the feasibility of friction stir welding process to join TiNi shape memory alloy sheets flat memorized, preserving the TiNi shape memory behaviour. This aim has been pursued determining the influence of the thermomechanical modifications induced by Friction Stir Processing of TiNi sheets on the functional properties of material. Optical microscopic investigations of Friction Stir Processed material cross sections have been used to highlight the modified microstructure of processed zone. A proper image processing procedure has been performed in order to quantify the amount of martensitic phase and to detect its morphology modification along the processed region. Particularly each micrographic image at first has been denoised using the 2D Wavelet transform technique and successively a texture segmentation procedure allows evaluating the amount of the martensite and austenite phases and classifying the morphological changes of martensitic regions. The austenitic and martensitic transformation temperatures of material were investigated using a stress applied characterization method suitably set up to perform the whole stress-temperature material characterization.

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Keywords: Wavelets image analysis; Friction Stir Processing; Shape memory alloys.

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Nomenclature

SMA	shape memory alloy
SME	shape memory effect
FSW	friction stir welding
FSP	friction stir processing
As	temperature which the austenitic transformation starts during heating
Af	temperature which the austenitic transformation finishes during heating
Ms	temperature which the martensitic transformation starts during cooling
Mf	temperature which the martensitic transformation finishes during cooling
VL	variable load
FI	functional index
DR	ductility reduction
SMR	shape memory reduction
SD	sinking depth
AR	as received

1. Introduction

Shape Memory TiNi alloys are functional materials able to return to a previously defined shape when subjected to an appropriate thermal cycle. During the fusion welding process of SMA, several undesired aspects as brittleness and losing of functional properties, must be taken into account. Laser welding is, at present, the best joining techniques for NiTi alloys and weld strength of about 75% of the raw material tensile strength may be achieved. The main disadvantage of this technique is due to the reduction in mechanical and shape memory performance of welded material and furthermore weld cracking may appear especially in the case of welding Ti-Rich shape memory alloys. The NiTi laser welded joints are therefore not suitable for the realization of smart components in constrained recovery applications, where a large strain must be recovered and a large tensile stress is induced. The instability of transformation cycles, that appears in term of variations in transformation temperatures, makes difficult the accurate predictions of materials behavior and makes inaccurate designing of components for SME applications. In order to overcome the cyclic instability problem it is necessary to increase the critical shear stress for dislocation slip. This goal, as reported in literature, is achievable by means of coherent nano-precipitation, grain refinement and strain hardening by dislocation substructures.

Friction stir welding is thus attractive for SMA joining and it exhibits potentials for the achieving of welded joints affected by microstructural changes that preserve the shape memory properties, the cyclic stability of the SME and the joint strength.

The basic objective of this study is to investigates the feasibility of friction stir welding process to join NiTi shape memory alloy sheets flat memorized preserving the NiTi shape memory behaviour. This aim has been pursued determining the effect of the FSP on the functional properties of material.

The effectiveness of friction stir welding process to join NiTi shape memory alloys was investigated by producing friction stir processed regions in thin sheets and by determining the effect of the high strains and the thermal cycles connected to the process on the functional properties of material. Metallographic analysis have been employed to highlight microstructural features of processed zone, furthermore, starting from the micrographic captures at 1000X of magnification, a two-step image analysis procedure has been set up in order to quantify the amount of the austenite and martensite phases and in order to detect also the morphology and the distribution characteristics of each phase. Particularly a wavelet based transform and a texture segmentation technique have been coupled with the aim to denoise all the micrographic images.

The austenitic and martensitic transformation temperatures and the functional behaviors of the processed material were determined using a characterization method based on a variable load test. The indexes FI, DR and SMR were defined in order to investigate the origin of the functional changes of material as result of the friction stir process. A post processing annealing treatment, following indicated as TT, has been performed in order to investigate the possibility to recover the partial losing of the shape memory capability subsequent to the FSP [1-7].

2. Variable load characterization, annealing and image processing experimental procedures

The shape memory material employed in this study was a Ti – 49.4Ni (at. pct.) thin sheet flat memorized, which keeps in the martensitic state at room temperature. The FSP was performed on two 100 mm × 70 mm thin sheets (0.5 mm) for a length of 40 mm. The processing parameters have been fixed equal to 500 rpm and 50 mm/min. The influence of the variation of the tool sinking depth (SD) was investigated by selecting two values of this parameter respectively equal to 0.2 mm and 0.3 mm. From each processed sheet were transversally cut specimen (100 mm × 8 mm) with the aim to highlight the microstructural occurrences on the cross section of the processed material and to perform the VL test. From the metallographic investigations of cross sectioned specimens it was possible to distinguish four typical FSW regions respectively named Nugget, Thermo Mechanical Affected Zone, Heat Affected Zone and Base Material. The shape memory properties were measured by a variable load test performed by employing a simple specifically designed equipment constituted by a clamping device for the specimen, a bending spike positioned to bend the friction stir processed region, a contrast spring in order to macroscopically detect the phase transformations during the cooling and a goniometrical reference in order to measure the bending angle variations. The contrast spring applies a variable load on the material during the thermal scanning as function of the spring elongation. For each bending angle it is possible to compute the corresponding load value, by recording the spring elongation, and therefore it is possible to define the load levels at which the transformation temperatures were measured. The VL tester was heater up to the temperature Af in order to activate the shape memory effect (SME). During the thermomechanical cycling, the temperature was measured using a K thermocouple and it was recorded together to the bending angle using a digital camcorder. The VL characterization tests highlighted that the FSP gives both ductility and shape memory capability reduction. In order to quantify these two different effects and to compare them to the functional properties of the base material three indicators, respectively named functional index (FI), ductility reduction (DR) and shape memory capability reduction (SMR), have been defined. Considering the initial bending angle θi of specimen and the final bending angle of specimen after shape recovery θf the FI is given by the expression (1):

$$FI\left[pct.\right]=\left\{\left[\frac{\left(\frac{\theta_i-\theta_f}{90}\right)}{0.8}\right]\times 100\right\} \tag{1}$$

in which 90 degrees is the initial bending angle of the base material and 0.8 is the effective shape memory capability of the AR material. The FI index therefore quantifies the total intrinsic material shape memory capability compared to that one of the AR material and considering, also, the decreasing of material ductility associated to the FSP. The indicator of percentage of the material ductility reduction (DR) is given by the expression (2):

$$DR\left[pct.\right]=\left\{\left[1-\left(\frac{\theta_i}{90}\right)\right]\times 125\right\} \tag{2}$$

In effect, when the parameter θi is equal to 90 degrees, and therefore the θi value is that one of base material, the DR is equal to 0 pct.; while, at increasing of the parameter θi the DR value increase also. The DR index foresees the total loosing of ductility properties when the θi value is equal to 18 degrees. The only percentage of the shape memory capability reduction (SMR) may be evaluated as the expression (3):

$$SMR\left[pct.\right]=\left\{\left[\left(\frac{\theta_f}{72}\right)-0.25\right]\times 100\right\} \tag{3}$$

In effect, when the parameter θ_f is equal to 18 degrees, and therefore the θf value is that one of base material, the SMR is equal to 0 pct.; while, at increasing of the parameter θf the SMR value increase also. The SMR index foresees the total loosing of shape memory properties when the θf value is equal to 90 degrees. The ductility reduction affects the total intrinsic shape memory capability of processed material and determines a reduction of the FI index value. The key point of this characterization method is the occurrence that the generated variable load is strictly correlated with the functional material. There is therefore the possibility to give simultaneously information of the transformation material temperatures and of the FSP effect the SMA functional properties. Following the FSP, the VL tests evidenced the occurrence of considerable reduction of the functional properties of processed material due to the reduction of both the shape memory and ductility properties. A significant recover of functional properties of material has been obtained by performing a post-processing annealing (TT) following the typical thermal cycle used in order to confer the shape memory properties to the NiTi alloys. The processed specimens, therefore, have been heated up to 723°K and maintained at this temperature for 300 seconds; finally, they have been quenched in cold water. VL tests and micrographic observations have been performed also on the post-thermal treated specimens.

Metallographic investigations of cross sectioned specimens were carried out using suitable mounting, grinding, polishing and etching phases. Particularly, mounted samples were ground using SiC paper with successively decreasing grit size respectively equal to 400, 600, 1000 and 1200. Samples were subsequently polished using 0.25 mm polycrystalline diamond and etched with a reagent composed by 14 ml HNO_3, 3 ml HF and 82 ml H_2O. The material was then observed by optical microscopy. The micrographic observations highlighted the typical friction stir welded cross sections region morphology of Ti alloys. The micrographic observations of FSP material highlighted the difficulty to easy distinguish and quantify the martensite and austenite phases in the regions affected by strong values of thermomechanical parameters and therefore in which the deformations and temperatures reached the higher values during FSP. This occurrence appears more significant in the nugget and TMAZ regions, but, at the varying of the thermal contribution, as a function of the sinking depth parameter, it may affects also the HAZ. A proper image processing technique has been performed on each micrographic image in order to quantify the amount of austenite and martensite phases in the characteristic regions of FSP material and to establish a mutual relation between the functional properties of SMA and the microstructural changes. Firstly the micrographic captures were subjected to a denoising procedure, using 2D stationary Haar wavelet transform at five level of decomposition, that allows to obtain on each image only two uniform regions for the austenite (smooth) and martensite (rough) phases; after, the denoised images were converted into grey scale images and enhanced by means of adaptive histogram equalization.

Finally the texture segmentation procedures, followed by image binarization, allowed to distinguish and quantify the amount of each phase. The whole image processing procedure is summarized in Figure 1. Both the wavelet image denoising and the texture based segmentation analysis were performed by means of the MATLAB® software. For each typical region of processed material, as Nugget, TMAZ, HAZ and base material, were captured and image processed five micrographic images at 1000X magnification. The obtained percentages of martensite or austenite phases were computed as mean of five values related to the previous mentioned micrographic captures.

3. Results and discussions

The functional characterization of friction stir processed TiNi alloys was developed both from a macroscopic point of view and from a microscopic point of view. Macroscopically the variation of the functional properties of material it has been assessed, respect to the ones of the base material, through the definition and the subsequent evaluation of three performance indices and also the variation in the transformation temperatures of material it has been assessed as a result of the friction stir process. All these mentioned parameters constitute the variable load tests results. From the microscopic point of view, an assessment of the percentage of retained austenite and martensite in the different regions characteristics related to the FSP process was carried out, in addition to identifying the distribution of the above mentioned phases within the same regions. Below, the characterization results of variable load tests are described starting from the transformation temperatures, in term of ΔLoad/ΔTemperature factors, that quantify the susceptibility of material's transformation temperatures to the variation of the load conditions. After, the obtained results in the phase of performance index evaluation and the obtained results from the micrographic image analysis phase are reported.

3.1. Transformation temperatures

In order to evaluate the phase transformation temperatures (M_s, M_f, A_s and A_f) under different loads for both as-received (AR or BASE) and processed (FSP) materials the VL tester was heated, by a resistance heater, up to the temperature A_f in order to activate the shape memory effect (SME).

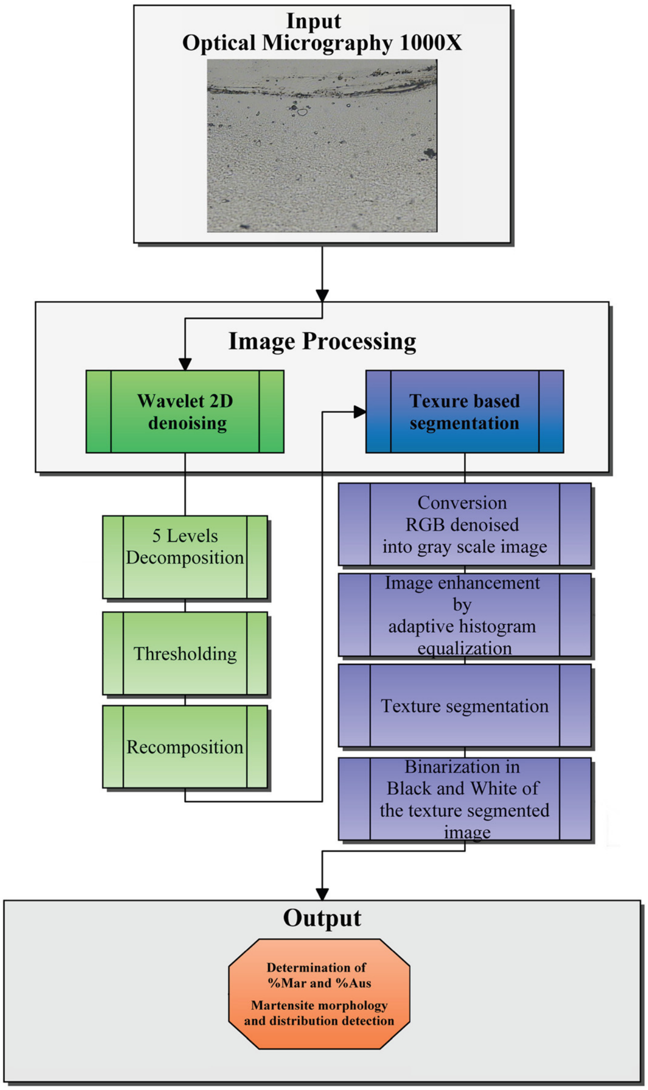

Fig. 1. Synthesis of the image processing procedure.

The cooling down under the M_f temperature was carried out using a water cooler. During the thermomechanical cycling, the temperature was measured using a K thermocouple and it was recorded together to the bending angle using a digital camcorder. Regarding the changes of transformation temperatures values, following the FSP of TiNi material, it has been observed that for lower thermal contribution, and therefore for the SD parameter value equal to

0.2 mm, there is a reduction of all the transformation temperatures compared whit those of the base material. Particularly it has been found a considerable reduction of M_s temperature (29°K) that indicates the presence of austenite stabilization phenomena. The post processing annealing treatment generates a reduction of the above mentioned phenomenon; particularly, the M_f and A_f temperatures are almost the same of the ones observed for the base material, while the M_s and A_s temperatures preserve a small reduction compared to the same transformation temperatures of the base material respectively equal to 7°K and 11°K. In the case of higher thermal contribution, and therefore for the SD parameter value equal to 0.3 mm, the FSP generates a small increasing of the A_f value (5°K) and an higher increasing of the M_s value (22°K) respect to the same temperatures of the base material. The post processing annealing treatment generates a considerable reduction of the M_s temperature respect the previous value. Particularly comparing the M_s temperature after post processing annealing whit the same temperature observed in the base material there is a reduction equal to 12°K.

3.2. *Susceptibility of transformation temperatures to vary under load*

It has been observed that, for lower values of heat input during the FSP process, and consequently, for value of sinking depth of 0.2 mm, there is a considerable increase of the variability of material's transformation temperatures values at the varying of the stress conditions with respect to what was observed for the base material in reference to the temperatures A_s, M_f, A_f and M_s. In effect, the ΔLoad/ΔTemperature factors are decreased, respect to the slope of A_s, M_f, A_f and M_s temperature for the base material, respectively of 37.5 (pct.), 37.5 (pct.), 39 (pct.) and 44 (pct.). The following post processing heat treatment, consisting in the annealing at 723°K, has the effect of returning the value of the susceptibility of transformation temperatures to the stress levels to the one observed for the base material for the temperatures A_s, M_f, and M_s. The ΔLoad/ΔTemperature factor for the A_f temperature, instead, increases of 91 (pct.) respect to the slope value observed for the same temperature line in the base material. High values of heat input during FSP lead to a considerable decrease of susceptibility of transformation temperatures to the existing stress levels compared to the detected susceptibility value for the base material. In effect the ΔLoad/ΔTemperature factors are increased respect to the slope of A_s, M_f and M_s temperature for the base material, respectively of 212 (pct.), 181 (pct.) and 183 (pct.), whereas, the ΔLoad/ΔTemperature factor for the A_f temperature line maintains the same value owned by the base material. As a result of the post processing annealing treatment, such reduction of susceptibility of transformation temperatures to the stress levels it attenuates, while maintaining the observed reduction of susceptibility value as compared to the base material. It is therefore noted that the material, subjected to FSP whit SD parameter equal to 0.3 and then thermally treated, possesses transformation temperatures characterized by a reduced variability under varying load conditions, compared to the variability of temperatures for the base material, for all the transformation temperatures. This property leads to the possibility to use a FSP component under varying load condition whit a small change of transformation temperatures considering the different working conditions.

3.3. Performance indexes evaluation

The evaluation of the performance indexes allowed to highlight that the FSP of material generates a remarkable reduction of the functional properties of material. Particularly, the functional index value, that quantify the amount of the functional properties of material preserved after FSP, indicates that the low thermal contribution determines the reduction of the functional properties of material equal to 72 (pct.) if compared to the functional properties of the base material. This functional reduction may split-up into two different contributions respectively attributable to the ductility reduction and to the shape memory capability reduction. Particularly it have been found that, when the thermal contribution during the process is lower, and therefore the SD parameter is equal to 0.2 mm, the two above mentioned contributions are respectively 14 pct. for the ductility reduction and 58 pct. for the shape memory capability reduction. The post processing annealing allowed to recover a part of the functional properties of material; in fact the FI value after annealing is equal to 44 pct. It may to remark that the performed thermal treatment was able to fully recover the ductility reduction of material, generated by FSP, but it was able to recover only a small part of the SMR. For process parameters able to produce higher thermal contribution, and therefore, for SD parameter equal to 0.3 mm, the reduction of functional properties, as a result of the FSP, was found more significant than the previous case; in fact the FI value was found equal to 16.7 pct., indicating therefore a substantial reduction of the functional characteristics of processed material. Regarding of the two different contributions, respectively attributable to the ductility reduction and to the shape memory capability reduction, were both found equal to 42 pct. The observed DR and SMR values suggested that higher thermal contribution is able to activate phenomena that promote the increasing of the DR parameter and the decreasing of the SMR parameter respect to the values of the same parameters obtained in the case of SD equal to 0.2 mm. The post processing annealing treatment, in this case, was able to fully recover the ductility reduction, and furthermore, it was able to recover up to 21.4 pct. in the shape memory reduction produced by FSP. Ultimately, for the specimen 0.3 SD TT, was found the possibility to preserve up to 66.7 pct. of the functional properties of material respect to the ones of the as received material. These described results, highlighted that the FSP, depending on the thermal contribution value, activates different solid state modification phenomena that influence the functional properties of material; particularly, there are some phenomena that compromise almost irreversibly the shape memory capability of material, and others that allow to recover a considerable amount of the functional properties of material by means of the post-processing annealing treatment. The discussion on these influencing factors was reported in the section 3.5.

3.4. Micrographic image analysis results

The image analysis phase, applied to the micrographic captures, allowed to quantify the amount of the austenite and martensite phases inside the characteristics Base, HAZ, TMAZ and Nugget regions of friction stir processed specimens. The obtained results underlined that, in the case of SD parameter equal to 0.2 mm, there are small regions of retained austenite inside the austenite matrix, with total extension that gradually decreases from the nugget, toward the TMAZ and finally toward the HAZ area. The amount of retained austenite inside the nugget attains, after FSP, the value of 38 pct. of total area, inside the TMAZ attain the value of 35 pct. of total and inside the HAZ attain the value of 4 pct. of total. The post-processing annealing treatment generated an increasing of the martensite percentage inside all the above mentioned regions, but the amount of this recover was decreasing from the nugget toward the HAZ. In effect, the post- processing thermal treatment promoted the reduction of 6 pct. of retained austenite inside the nugget, the reduction of 3 pct. of retained austenite inside the TMAZ and the reduction of 1 pct. of retained austenite inside the HAZ area. In the case of SD parameter equal to 0.3 mm, they were found inside the NUGGET and TMAZ areas some regions of retained austenite; besides, the decreasing in the martensite amount has been more noticeable in the Nugget with respect to the TMAZ area. The post-processing annealing treatment generated a considerable increasing of the martensite percentage inside all the above mentioned regions and the amount of this recover was decreasing from the nugget toward the TMAZ. In effect, the post- processing thermal treatment promoted the reduction of 14 pct. of retained austenite inside the nugget and the reduction of 8 pct. of retained austenite inside the TMAZ area. The obtained results underlined that, depending on the thermal contribution value, the FSP is able to activate different solid state modification phenomena that they influence the content of martensitic and austenitic phases in each characteristic area of processed material. Particularly, there are some phenomena that generated austenite stabilization areas retained after annealing treatment, while, others solid state modifications allow to promote another austenite stabilization process that it is significantly attenuated by means of the annealing treatment. Finally, regarding of the martensite morphology, it was observed that inside the regions affected by plastic flow of material, during FSP, there is a preferential orientations of martensite phase and furthermore, inside the Nugget, the balance between the deformation level and the activated DRCX phenomenon promotes the development of small plates of preferential oriented martensite.

3.5. Influential factors

All the described results were determined by different influential factors activated during the FSP or the prost processing annealing. These factors may be resumed into:

- Factors affecting the Ductility of material;
- Factors affecting the Shape Memory capability of material.

A relevant reduction of material ductility was observed in all the specimen subjected to FSP, but this reduction was totally recovered for all the used process parameters by the post processing annealing treatment. These considerations, coupled whit the micrographic observations, they suggested that the DR phenomenon is attributable to the preferential orientation of martensite phase inside the Nugget and TMAZ regions. The effect of the post processing annealing treatment allows to obtain a reorientation of the martensite variants that, now, may develop themselves according to all possible orientations. This reorientation of variants guarantees the recovering of the ductility reduced after the FSP. The Shape Memory capability of material is influenced by the amount of the retained austenite developed during the FSP. Were highlighted two different mechanisms that determine the austenite stabilization: the first one is directly connected to the ausforming of material during the process and the second one is related to the aging of material that may appear when the thermal contribution, determined by the SD parameter, maintains the process temperature below 1100°K. In effect, the ausforming of TiNi material produces an increased resistance to the movement of the austenite-martensite interface due to the dislocations density at the interface between the two phases. This phenomenon, more active where the CDRX is contained, makes stable the austenite phase at the room temperature. Depending on the SD parameter value, 0.2 mm or 0.3 mm, the thermal contribution during the FSP may determine the formation of the intermediate TiNi phase or may promote the activation of Ti_2Ni intermetallic precipitation in the case of lower thermal contribution. This aging Ti_2Ni precipitation constitutes another mechanism that prevents the martensite development inside the austenite regions, and determines, therefore, the stabilization of austenite phase. The post processing annealing treatment acts differently depending on whether the austenite is stabilized due to the ausforming or the aging. Particularly, the retained austenite generated by ausforming may be totally recovered, and therefore transformed into martensite phase, after the annealing treatment, while the retained austenite originated by aging remains also after the annealing treatment.

4. Conclusions

In this study the effect of Friction Stir Processing of TiNi material on their functional properties it has been investigated in order to evaluate the feasibility of friction stir welding process to join thin sheets of TiNi shape memory alloy. The functional characterization of friction stir processed TiNi alloys was developed both from a macroscopic and from a microscopic point of view. A characterization method able to provide the whole stress-strain-temperature characterization of the shape memory material in only one test has been proposed. A post processing annealing treatment allowed to investigate the possibility to recover the partial losing, experimentally found, of the shape memory and of the ductility of processed material. A structured image processing procedure of the micrographic captures of processed material, based on the 2D wavelets transformation and on the texture segmentation, it has been defined in order to quantify the amount of austenite and martensite phases and to establish the correlation between the functional properties of processed SMA and the observed microstructural changes. The most relevant obtained results are following summarized:

- A relevant reduction of material ductility was observed in all specimens subjected to FSP, but this reduction was totally recovered by the post processing annealing treatment.
- The Shape Memory capability of material is much influenced by the amount of the retained austenite developed during the FSP. Two different mechanisms that determine the austenite stabilization were highlighted: the first one is directly connected to the ausforming of material during the process and the second one is related to the aging of material that may appear when the thermal contribution, determined by the SD parameter, maintains the process temperature below 1100°K.
- The retained austenite generated by ausforming may be totally recovered, and therefore transformed into martensite phase, after the annealing treatment, while the retained austenite originated by aging remains also after the annealing treatment.
- The Friction Stir Processed material, using a SD parameter equal to 0.2 mm, evidenced that the retained austenite regions, found in the Nugget, TMAZ and HAZ areas, are almost totally retained after post processing annealing and this occurrence determined an high value of SMR parameter also after the annealing treatment.
- The Friction Stir Processed material, using a SD parameter equal to 0.3 mm, the best case, evidenced that the retained austenite regions, found in the Nugget and TMAZ areas, are considerably reduced after post processing annealing specially in the Nugget zone in which the retained austenite after annealing has been halved and

therefore, in this case, because of the higher value of the thermal contribution during the process, the predominant austenite stabilization phenomenon is related to the ausforming effect and the ductility reduction mechanism is also activated. Using these process parameters, the possibility to preserve, after FSP and post processing annealing, up to 66.7 pct. of the functional properties of material respect to the ones of the as received material has been found.

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